

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants: Cunningham et al.  
Serial No.: 09/543,612  
Filed: April 5, 2000  
For: APPARATUS AND METHOD  
FOR MEASURING THE MASS  
OF A SUBSTANCE

Confirm. No.: 6510  
Group: 2856  
Examiner: Chapman Jr., John E.  
Docket No.: DR-308J

AFFIDAVIT UNDER 37 CFR SECTION 1.132

I, Charles Spangler, hereby say:

I am an employee of the RJ Lee Group, Inc., which has taken a license from The Charles Stark Draper Laboratory, Inc. for the above-identified patent application. In particular, the RJ Lee Group, Inc. took a license for this patent application because the subject invention provides the ability to measure Non-Volatile Residue (NVR) concentrations at the nanogram and the sub-nanogram level and because the invention is able to operate significantly faster than the prior art devices. The RJ Lee Group, Inc. manufactures the Nanoscale 9100<sup>TM</sup>, a brochure of which is attached hereto, which embodies the subject invention.

After the RJ Lee Group, Inc. began manufacturing the Nanoscale 9100<sup>TM</sup>, it won the Clean Technology Award of 2002, a copy of which is attached hereto. This award clearly shows that the subject invention is not only novel, but innovative as well.

Since the RJ Lee Group, Inc. began manufacturing the Nanoscale 9100<sup>TM</sup> there has been incredible market demand for this product. Twenty-three different companies have made separate inquiries about purchasing the Nanoscale 9100<sup>TM</sup> thereby showing a long-felt need for a product of this type. Among the well known companies that have inquired about purchasing the Nanoscale 9100<sup>TM</sup> are the Kennedy Space Center, the Stennis Space Center, Boeing, Boeing Commercial Aircraft, Rocketdyne, the Crane Division of the Naval Surface Warfare Center, the Los Alamos National Laboratory, Procter & Gamble, the Lawrence Livermore National Laboratory, Eli Lilly and Company, and Pfizer. Many of these companies independently contacted the RJ Lee Group, Inc. about the Nanoscale 9100<sup>TM</sup> after hearing about the product.

Prior art devices could perform NVR measurements, but these devices could take several hours to complete a measurement that the subject invention could perform in minutes. To my knowledge, there has been a failure of other companies to bring to the market a comparable product that offers the advantages of the Nanoscale 9100<sup>TM</sup>.

The RJ Lee Group, Inc. has agreed to pay a substantial license for the subject invention which includes paying a royalty for each individual sale of the product associated with the subject invention.

As described in detail below, there is a clear nexus between the claims of the above-referenced patent application and the economic success of the Nanoscale 9100™.

Claim 26 of the subject invention recites, "A method of measuring the concentration of particles in a solution, the method comprising: depositing a measured quantity of the solution on a sensor having a membrane layer; allowing the solution to evaporate until the particles remain on the membrane layer; driving the membrane layer at a reference resonant frequency; detecting the shift in frequency of the membrane layer due to the mass of the particles; determining the mass of the particles based on the shift in frequency; and based on the measured quantity of the solution and the mass of the particles, automatically calculating the concentration of the particles in the solution." Independent claim 27 recites features similar to claim 26 but relates to the use of a flexure plate wave sensor. Independent claims 28 and 30 relate to concentration detection systems that include either a sensor having a membrane layer or a flexural plate wave device, respectively.

U.S. Patent No. 5,212,988 to White et al. fails to teach, disclose or suggest allowing a solution to evaporate until particles remain on a membrane layer of a sensor or on a flexure plate wave sensor, as recited in independent claims 26 and 27, respectively.

White et al. also fails to teach, disclose or suggest automatically calculating the concentration of the particles in the solution based on the measured quantity of the solution and the mass of particles, in which the mass of particles is determined from detecting the shift in frequency of a membrane layer or a flexure plate wave sensor due to the mass of the particles, also recited in independent claims 26 and 27, respectively.

U.S. Patent No. 5,918,258 to Bowers relates to the use of SAW sensing devices and thus fails to disclose the use of a sensor that includes a membrane layer or a flexure plate wave sensor. Similar to White et al., Bowers also fails to teach, disclose or suggest the features of independent claims 26 and 27, specifically, allowing a solution to evaporate until particles remain on a membrane layer of a sensor or flexure plate wave sensor; or calculating the concentration of the particles in the solution based on the measured quantity of the solution and the mass of particles, in which the mass of particles is determined from detecting the shift in frequency of a membrane layer or a flexure plate wave sensor due to the mass of the particles.

Also, both White et al. and Bowers fail to teach, disclose or suggest the features of independent claim 28, specifically, a concentration detection system that includes a sensor having a membrane layer, a transducer for detecting the change in frequency of the membrane layer due to the particles after a solution evaporates; and a processor configured to automatically determine the mass of the particles based on the change in frequency, and to calculate the concentration of the particles in the solution based on the mass of the particles and the quantity of the solution deposited. Independent claim 30 includes similar features and is directed to a flexure plate wave sensor.

There is a clear nexus between the economic success of the Nanoscale 9100™ described above and the claims of the above-referenced patent application. The Nanoscale 9100™ has achieved economic success due to the stated differences between it and the prior art. As evidence of this, the differences between the prior art and the claimed invention are clearly promoted in the enclosed brochure for the Nanoscale 9100™, thus showing a significant nexus between the subject patent application and the commercial significance of the Nanoscale 9100™.

Specifically, page 1 of the enclosed brochure for the Nanoscale 9100™ states that:

[t]he Nanoscale 9100 is a simple, fast and inexpensive method for measuring [nonvolatile residue] concentration in any liquid sample using a minimal sample volume. With its built-in heater the gravimetric sensor is continuously temperature stabilized and the time required for evaporation is minimized.

(Emphasis added.) Page 2 of the same brochure expands upon the commercial significance of the Nanoscale 9100™ sensor:

**NanoScale 9100 - Unique Sensor Technology**

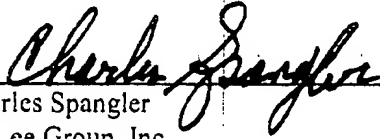
The [Flexure Plate Wave] sensor operates as a mass detection device by registering a decrease in the resonant frequency of a thin silicon membrane when mass is deposited on its surface. Because the resonant frequency shift is proportional to the amount of deposited mass, the sensor can be used to quantify the amount of material on the surface. The accuracy is determined by the sensors [sic] inherent sensitivity and by the frequency resolution of the electronic circuit used to drive the resonant mode.

(Emphasis added.)

Thus, the attached brochure makes clear that the commercially significant aspects of the Nanoscale 9100™ closely correspond to the features of the subject invention as claimed.

Also, there is a nexus between the stated objects of the subject invention and the commercial success of the Nanoscale 9100™. One of the objects of the invention includes providing an apparatus and method that is capable of determining the mass of a substance with a resolution in the nanogram range, and that is capable of determining the mass of a substance in a quick and simple manner. It is another object of the invention to monitor the change in the reference resonant frequency of a flexural plate wave sensor to determine the mass of a substance disposed on the sensor. See the subject patent application at page 3, lines 6-18. Features corresponding to these objectives are clearly mentioned in the enclosed brochure for the Nanoscale 9100™, thus showing a clear nexus between the subject patent application as claimed and the commercial significance of the Nanoscale 9100™.

The undersigned, being hereby warned that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 USC 1001, and that such willful false statements may jeopardize the validity of the application or any resulting registration, declare that the facts set forth in this declaration are true; all statements made of his own knowledge are true; and all statements made on information and belief are believed to be true.

  
Charles Spangler  
RJ Lee Group, Inc.

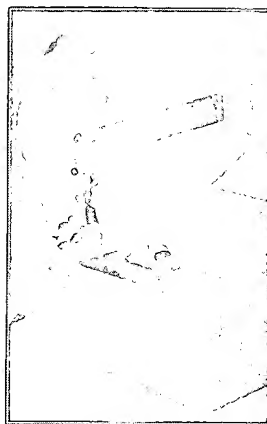
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Date

# Revolutionizing Gravimetric Measurement

## NanoScale 9100

Analyze Non-Volatile Residues with nanogram sensitivity from only 10 microliters of sample in just minutes... anywhere!

The NanoScale 9100 is a novel system for detecting and quantifying Nonvolatile residue (NVR) concentrations in liquids using a micromechanical flexural plate wave (FPW) gravimetric sensor. It can also be used to

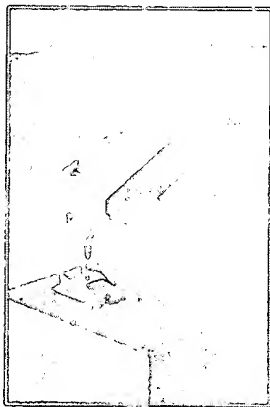


Lead sample.

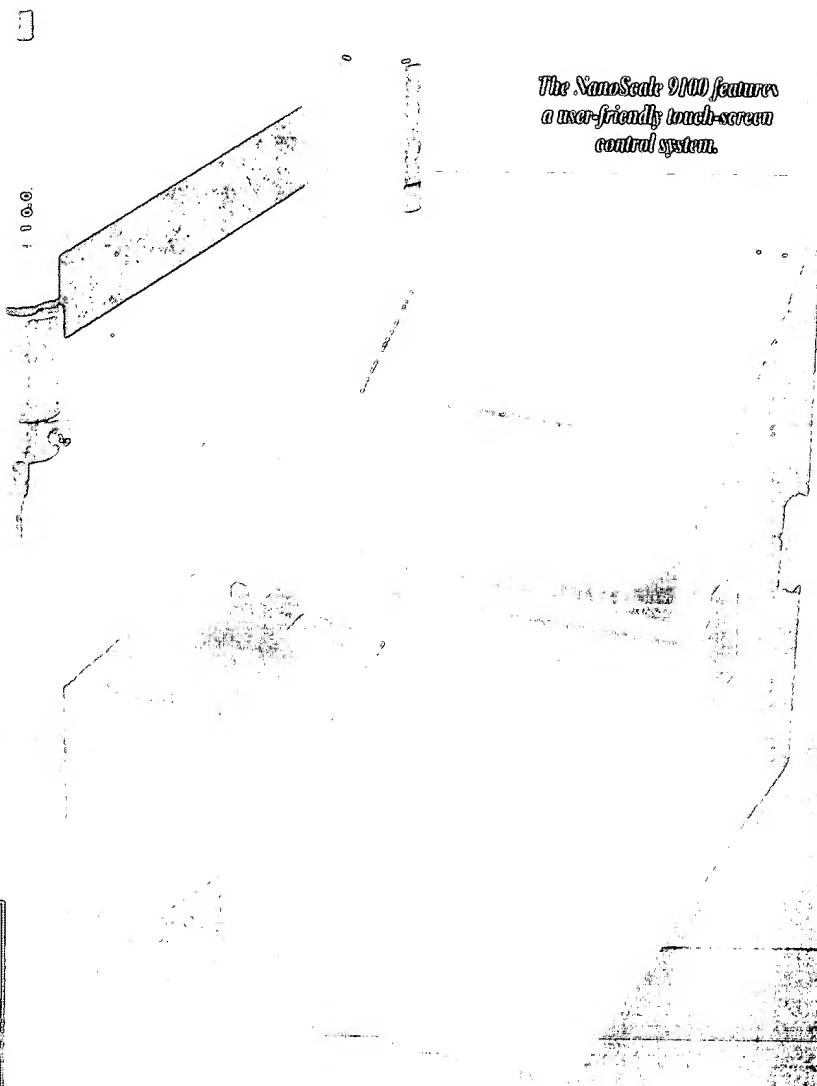
measure any small, uniformly deposited film for microliter syringe calibration, solution concentration measurements, aerosol distribution, etc. There is a growing need to measure the purity level of many chemicals both during their manufacture and use. In particular, the level of nonvolatile contaminants are generally of interest in high-purity solvents because they will remain as residual contaminate after the solvent has been used in a cleaning process.

For typical solvents, the NVR to be measured will be on the order of 0.1 to 100 ppm in concentration and requires the evaporation of large quantities of the solvent, tight environmental control of the measurement area, and lengthy procedures to ensure the needed accuracy. Quantification of NVR concentration is critical for many processes in precision cleaning, semiconductor, pharmaceutical, chemical and food processing industries where high-purity chemical reagents are required. For these industries, NVR qualification testing is required by quality assurance programs for all externally supplied reagents used in critical operations.

The NanoScale 9100 is a simple, fast and inexpensive method for measuring NVR concentration in any liquid sample using a minimal sample volume. With its built-in heater the gravimetric sensor is continuously temperature stabilized and the time required for evaporation is minimized.



Inject sample.



The NanoScale 9100 features a user-friendly touch-screen control system.

### NanoScale 9100 - The Lab On A Chip

- Only 10 microliters of sample fluid required
- NVR measurement in as little as 5 minutes
- Nanogram sensitivity (i.e. <1ppm NVR)
- Continuously temperature stabilized
- Rugged - does not require enclosure or vibration isolation
- Direct readout in mass (nanograms) or concentration (ppm/ul)
- Small footprint by using MEMS technology
- Reduces total laboratory emissions

# NanoScale 9100

## Applications

The NanoScale 9100 can be used for a variety of weighing applications where ultra-high sensitivity, speed and simplicity are desired including:

- Measurement of Non-Volatile Residues (NVR) in solvents
- Cleanliness analysis of water discharged from semiconductor wafer processing
- Incoming inspection of high purity reagents and solvents.
- Quality control in production of high purity chemicals
- Monitoring of fine aerosols in painting and coating operations
- Measurement of non-volatile precipitates in high purity water for cleaning and processing
- Monitoring of fine particulate in ambient air and fluids
- Measuring active ingredients in a volatile carrier (pharmaceuticals)

## Disadvantages of Current NVR Measurement Techniques

Standard laboratory practice is to boil a large quantity (up to a liter) of solvent sample until the entire sample is evaporated. Any material remaining after boiling is nonvolatile residue (NVR). A microbalance is used to measure the weight of any material remaining after boiling. A large quantity of solvent must be boiled away in order to provide enough mass gain to be resolved by the microbalance. Typically, NVR concentrations of interest are in the 0.1-100 ppm range.

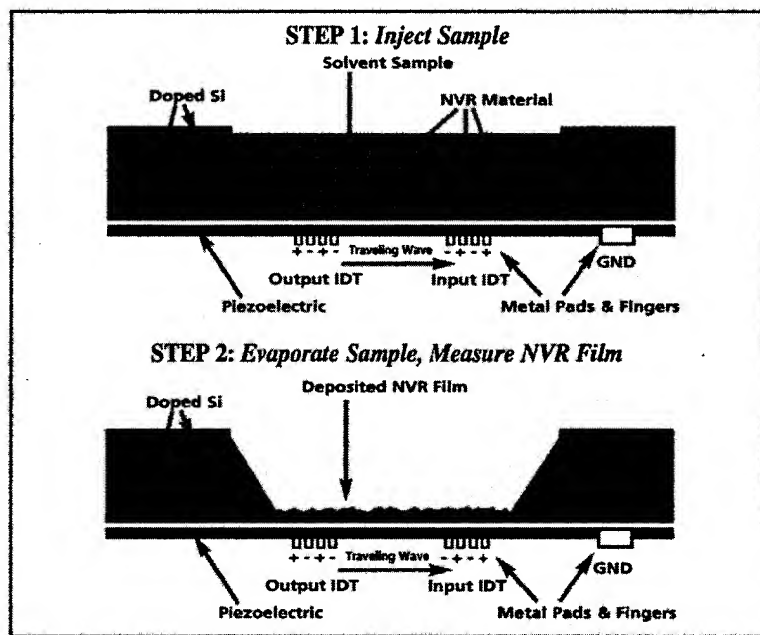
Disadvantages of this method include: high volumes of solvent are consumed; the boiling and measuring operations take several hours; boiled solvent is exhausted into the atmosphere; measurement process is labor intensive; reproducibility problems arise due to the ease of contamination during long sample exposure times.

## NanoScale 9100 - Unique Sensor Technology

The FPW sensor operates as a mass detection device by registering a decrease in the resonant frequency of a thin silicon membrane when mass is deposited on its surface. Because the resonant frequency shift is proportional to the amount of deposited mass, the sensor can be used to quantify the amount of material on the surface. The accuracy is determined by the sensors inherent sensitivity and by the frequency resolution of the electronic circuit used to drive the resonant mode.



*Automated Analysis Mode*



*Schematic View of Sensor*



*Suggested Retail Price: \$16,500 Domestic, \$18,000 International*

**RJ Lee microsystems**  
LLC

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# CleanTech™

For Cleaning Process Improvement

Charlie Simpson  
Editor  
CleanTech magazine  
84 Park Avenue  
Flemington, NJ 08822  
April 30, 2002

Robert J. Lee  
President  
RJ Lee Group  
350 Hochberg Road  
Monroeville, PA 15146

Mr. Lee,

The Cleaning Technology Awards recognize technology innovations equally across both the industrial and precision cleaning markets. From the numerous nominations submitted for the 2002 Awards, CleanTech's staff selected 15 qualifying candidates. CleanTech's editors invited our readers to help us decide this year's most pioneering technologies, by submitting ballots featured in the magazine.

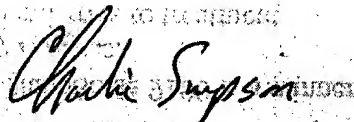
It is my pleasure to inform you that RJ Lee Group's NanoScale 9100 is a winner of CleanTech magazine's third annual Cleaning Technology Awards.

Ranging from outsourced services and alternative chemistries to treatment hardware and complete wash systems, this year's nominations exemplified how the cleaning industry remains diverse and growing.

These products help to improve production, eliminate bottlenecks, comply with regulatory mandates, reduce costs, increase safety, and of course, improve the cleanliness process – all critical issues for any manufacturer.

From those, the NanoScale 9100 was chosen as an outstanding technology that the CleanTech staff believes offers revolutionary benefits to our readers.

The 2002 Cleaning technology trophies will be presented at the Awards Breakfast at the CleanTech 2002 Conference and Expo being held in Atlanta's Cobb Galleria Centre, May 21 – 23, 2002 at our Awards Breakfast on Wednesday May 22, at 8:00 am. I invite you and your staff to attend this year's ceremony. Congratulations, and I look forward to seeing you at the awards presentation in Atlanta.

  
Charlie Simpson  
Editor



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